



# Kongeriget Danmark

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**Patent- og  
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Modtaget

**TITEL:** PARTICLES CONTAINING A ELASTIC COATING**FIELD OF THE INVENTION**

The present invention relates to coating materials for coating  
5 particles containing an active. Said coating materials are  
elastic and light and serve both the purpose of regulating  
density of the particles a protects the particle from damage by  
being able of absorbing impacts in the particle. The invention  
also relates to processes for the manufacture of such coated  
10 particles and to the use of such coated particles in a number of  
applications.

**BACKGROUND OF THE INVENTION**

It is known to the art to incorporate actives such as enzyme into  
dry solid particles and thereby protect the active from  
15 inactivation and/or protect the environment form the active. Such  
products have usually been applied to other dry products such as  
dry granular detergents compositions to improve their  
performance. Such particles containing an active is prepared by  
applying the active to a mixture of solids. Among actives which  
20 suitably may be incorporated in dry solid particles are enzymes.

Known enzyme granule formulation technologies include:

a) Spray dried products, wherein a liquid enzyme-containing  
25 solution is atomised in a spray drying tower to form small  
droplets which during their way down the drying tower dry to  
form an enzyme-containing particulate material. Very small  
particles can be produced this way (Michael S. Showell (editor);  
*Powdered detergents*; Surfactant Science Series; 1998; vol. 71;  
30 page 140-142; Marcel Dekker).

b) Layered products, wherein the enzyme is coated as a layer around a pre-formed inert core particle, wherein an enzyme-containing solution is atomised, typically in a fluid bed apparatus wherein the pre-formed core particles are fluidised, and the enzyme-containing solution adheres to the core particles and dries up to leave a layer of dry enzyme on the surface of the core particle. Particles of a desired size can be obtained this way if a useful core particle of the desired size can be found. This type of product is described in e.g. WO 97/23606

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c) Absorbed core particles, wherein rather than coating the enzyme as a layer around the core, the enzyme is absorbed onto and/or into the surface of the core. Such a process is described in WO 97/39116.

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d) Extrusion or pelletized products, wherein an enzyme-containing paste is pressed to pellets or under pressure is extruded through a small opening and cut into particles which are subsequently dried. Such particles usually have a considerable size because of the material in which the extrusion opening is made (usually a plate with bore holes) sets a limit on the allowable pressure drop over the extrusion opening. Also, very high extrusion pressures when using a small opening increase heat generation in the enzyme paste, which is harmful to the enzyme. (Michael S. Showell (editor); *Powdered detergents*; Surfactant Science Series; 1998; vol. 71; page 140-142; Marcel Dekker)

e) Prilled products or, wherein an enzyme powder is suspended in molten wax and the suspension is sprayed, e.g. through a rotating disk atomiser, into a cooling chamber where the droplets quickly solidify (Michael S. Showell (editor); *Powdered detergents*; Surfactant Science Series; 1998; vol. 71; page 140-142; Marcel Dekker). The product obtained is one wherein the

enzyme is uniformly distributed throughout an inert material instead of being concentrated on its surface. Also US 4,016,040 and US 4,713,245 are documents relating to this technique

5 f) Mixer granulation products, wherein an enzyme-containing liquid is added to a dry powder composition of conventional granulating components. The liquid and the powder in a suitable proportion are mixed and as the moisture of the liquid is absorbed in the dry powder, the components of the dry powder  
10 will start to adhere and agglomerate and particles will build up, forming granulates comprising the enzyme. Such a process is described in US 4,106,991 (NOVO NORDISK) and related documents EP 170360 B1 (NOVO NORDISK), EP 304332 B1 (NOVO NORDISK), EP 304331 (NOVO NORDISK), WO 90/09440 (NOVO NORDISK) and WO  
15 90/09428 (NOVO NORDISK). In a particular product of this process wherein various high-shear mixers can be used as granulators, granulates consisting of the enzyme, fillers and binders etc. are mixed with cellulose fibres to reinforce the particles to give the so-called T-granulate. Reinforced particles, being more  
20 robust, release less enzymatic dust (*vide infra*).

Known enzyme formulations are to day either "dry formulations", i.e. consisting of agglomerates of solid particles or "liquid formulations", i.e. liquids containing solutions or suspensions of enzymes/enzyme particles.

## 25 SUMMARY OF THE INVENTION

An object of the present invention is to provide improved coatings the particles containing an active, such as an enzyme. When preparing particles containing an actives one object is to separate them from their surroundings until the moment when they  
30 are to be used in an application. his has generally been achieved by incorporating the active in discrete particles. Another object for incorporating the active in a particle is to

lower the amounts of potentially harmful active dust which may be generated from the active.

Further we have found that as particles normally are sold by the amount of activity per weight of preparation it is  
5 desirable to apply light coatings, as the coatings which normally does not contain the active should not account for too much weight and the active should account for the majority of weight to provide high active contents particles.

Accordingly an object of the invention is to provide a  
10 lights weight coating which lowers the dust formation from the coated particles and at the same time the coating should be light in order to lower the overall true density of the coated particles.

The present invention provides a coated particle comprising  
15 a coating and a core particle comprising an active, wherein the coating comprises a gas phase component, which posses these desired properties.

The invention also provides methods for preparation of the coated particles and compositions comprising the coated  
20 particles and their use.

#### BRIEF DESCRIPTION OF THE TABLES AND DRAWING

No drawings

#### 25 DETAILED DESCRIPTION OF THE INVENTION

When handling solid particles comprising an active, one of the major problems is the formation of dust from the active, which may be harmful to persons handling the dry solid composition.

Although the active may be incorporated in dry solid  
30 particles as known to the art, which may inhibit the formation of active dust it is a fact that active dust are still being

released from known particles, e.g. as measured by the known Heubach Method, and there is a need for further improvement.

Conventional particles are usually solid and brittle which are susceptible to damage when a strain is applied to them. However by applying the elastic coating of the invention around the particle a mechanical elastic buffer is put between the particle and the environment causing strain on the particle.

### Definitions

The term "true density" of a compound as used herein, is to be construed as the density in weight per volume of said compound, determined by immersing a weighed amount of the compound in a liquid in which the compound is insoluble and measuring the volume increase of liquid dispersion (i.e. the volume of liquid which is displaced by the compound). As an example, if 1 gram of a compound is added to a volume of 10 cm<sup>3</sup> of a liquid in which the compound is insoluble and by said addition the volume of the liquid-compound mixture increases to 11 cm<sup>3</sup>, the compound thereby displacing 1 cm<sup>3</sup> of liquid, the true density of the compound is 1 gram per cm<sup>3</sup>. The true density of a liquid may be measured as the weight of a measured volume of the liquid.

The term "wax" as used herein, is to be construed as a compound having a melting point between 25-150 °C.

The phrase "ratio between the diameter of the coated particle and the diameter of the core particle" (hereinafter abbreviated  $D_T/D_C$ ) as used herein is to be understood as the diameter of a particle comprising a core particle and a coating divided by the diameter of the core particle only. If for example a core particle having a diameter of 100  $\mu\text{m}$  is coated with a coating layer 200  $\mu\text{m}$  thick, the coated particle would have a diameter of  $(200+100+200)=500$   $\mu\text{m}$  and  $D_T/D_C$  is  $500 \mu\text{m}/100 \mu\text{m} = 5$ .

The term "substantially enzyme free " as used herein about a coating means that there less than 5 mg of enzyme per gram coating.

#### **The coating**

5 The coating of the invention comprises a gas phase component. We have found that by incorporating a gas phase component, the density of the coating is lowered and at the same time by having a compressible gas phase in the coating to elasticity is improved. Preferably the coating matrix incorporating the gas  
10 phase component is a solid phase material.

The gas component is to be understood as any gas or mixture of gases, such as atmospheric air, carbon dioxide, nitrogen, noble gases.

The gas should constitute at least 20% by volume (v/v) of  
15 the coating material, preferably at least 40% by volume, more preferably at least 60% by volume and most preferably at least 80% by volume.

The gas comprised in the coating may be present in the form of gas bubbles entrapped in a matrix of other coating materials.  
20 It may also be entrapped in light spheres.

#### Light spheres

Light spheres are small particles with low true density. Typically, they are hollow spherical particles with air or gas inside. Such material are usually prepared by expanding a solid  
25 material. These light spheres may be inorganic of nature such as Scotchlite™ Glass Bubbles from 3M™ (hollow glass spheres), Q-CEL® (hollow microspheres of borosilicate glass) and/or Extendspheres® (ceramic hollow spheres) available from The PQ Corporation. The light spheres may also be of organic nature  
30 such as the PM-series (plastic hollow spheres) available from The PQ Corporation. Expancel® (hollow plastic spheres) from AKZO Nobel, Luxsil® and Sphericel® from Potters Industries and/or

Styrocell<sup>R)</sup> from SHELL, which is spheres of polystyrene. The polystyrene of Styrocell<sup>R)</sup> contains pentane which upon heating boils and expands or pops the material (the reaction is comparable to the expansion of corn seeds into popcorn) leaving  
5 a light polystyrene material of a low true density. Also polysaccharides are preferred, such as starch or derivatives thereof. Biodac<sup>®</sup> is an example of non-hollow light weight material made from cellulose (waste from papermaking), available from GranTek Inc. These materials may be included in the  
10 granules of the invention either alone or as a mixture of different light materials.

#### Other coating materials

The coating also contain a matrix of materials suitable for entrapping the gas phase component. Such materials includes but  
15 is not limited to waxes, polypeptides, and carbohydrate polymers, which may form an elastic network entrapping the gas.

The wax included in this list encompass polymeric compounds, which have a melting point between 20-150 °C. Preferred waxes are organic compounds or salts of organic  
20 compounds having a melting point in the said range. In the context of the invention the term "wax" as used herein also encompasses mixtures of two or more different waxes. Mixture of different waxes. Also, an important feature of the wax or mixture of waxes is that the wax should be water soluble or  
25 water dispersible, preferably in neutral and alkaline solution, so that when the coated particles of the invention is introduced into an aqueous solution, i.e. by diluting it with water, the wax should disintegrate and/or dissolve providing a quick release and dissolution of the active incorporated in the core  
30 particles to the aqueous solution. Examples of water soluble waxes are poly ethylene glycols (PEG's) Accordingly amongst water soluble waxes the solubility of wax in water should preferably be up to 75 parts wax to 25 parts water, such as for



PEG 1000. Amongst water insoluble waxes which are dispersible in an aqueous solution are triglycerides and oils.

The wax should be in a solid state at room temperature (25°C), and accordingly it has a melting point or a melting range (polymer waxes tend to melt over a range of temperatures) above this temperature. A preferred wax has a melting point or range between about 35°C to about 120°C. The lower limit is preferred to set a reasonable distance between the temperature at which the wax melts to the temperature at which liquid compositions comprising the particles are usually stored (20-30°C). Also, difficulties are contemplated, in the manufacture of the particles when the melting point of the wax is below 35°C. A more preferred melting point or range is between about 40°C to about 100°C, such as between about 50°C to about 80°C. In a further preferred embodiment the wax should have a molecular weight between about 150 Daltons to about 10,000 Daltons.

The wax of the invention may be any wax, which is chemically synthesized. It may also equally well be a wax isolated from a natural source or a derivative thereof. Accordingly in the wax of the invention is preferably selected from the following non limiting list of waxes.

- Poly ethylene glycols, abbreviated PEG, type of wax. Different PEG waxes are commercially available having different molecular sizes, wherein PEG's with low molecular sizes also have the lowest melting points. Examples of suitable PEG's are PEG 1500, PEG 3000, PEG 4000, PEG 6000, PEG 9000 e.g. from BASF - Germany. To meet the desired properties of true density and melting point for the wax and/or the enzyme particle, it is also contemplated that mixtures of waxes with low melting point with waxes of a high melting point is a very useful embodiment of the invention.

- polypropylenes or polyethylenes or mixtures thereof.
- Nonionic tensides which are solid at room temperature such  
5 as ethoxylated fatty alcohols having a high level of  
ethoxy groups such as Lutensol AT80 from BASF having 80  
units of ethyleneoxide per molecule. Alternatively  
polymers of ethyleneoxide, propyleneoxide or copolymers  
thereof are useful, such as in block polymers, e.g.  
10 Pluronic PE 6800 from BASF Germany.
- Waxes isolated from a natural source, such as Carnauba wax  
(melting point between 80-88°C), Candelilla wax (melting  
point between 68-70°C) and bees wax. Other natural waxes  
15 or derivatives thereof are waxes derived from animals or  
plants, e.g. of marine origin. Examples of such waxes are  
hydrogenated ox tallow, hydrogenated palm oil,  
hydrogenated cotton seeds and/or hydrogenated soy bean  
oil, wherein the term "hydrogenated" as used herein is to  
20 be construed as saturation of unsaturated carbohydrate  
chains, e.g. in triglycerides, wherein carbon-carbon  
double bonds are converted to carbon-carbon single bonds.  
An example hydrogenated palm oil is commercially available  
e.g. from Hobum Oele und Fette GmbH - Germany or Deutsche  
25 Cargill GmbH - Germany.
- Fatty acid alcohols, such as the linear long chain fatty  
acid alcohol NAFOL 1822 (C<sub>18, 20, 22</sub>) from Condea Chemie GMBH  
- Germany, having a melting point between 55-60°C and  
30 having a true density of about 0.96 g/cm<sup>3</sup>.
- Mono-glycerides and/or di-glycerides, such as glyceryl  
stearate, wherein stearate is a mixture of stearic and  
palmitic acid are useful waxes. An example of this is

Dimodan PM - from Danisco Ingredients, Denmark - having a true density of about 1 g/cm<sup>3</sup>

- Fatty acids, such as hydrogenated linear long chained fatty acids.
- Paraffines, i.e. solid hydrocarbons.
- Micro-crystalline wax.

10

In further embodiments waxes which are useful in the invention can be found in C.M. McTaggart et. al., Int. J. Pharm. 19, 139 (1984) or Flanders et.al., Drug Dev. Ind. Pharm. 13, 1001 (1987) both incorporated herein by reference.

15

Among polypeptides materials selected from gelatine, collagen, casein, chitosan, poly aspartic acid and poly glutamic acid are preferred

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Among carbohydrate polymers materials selected from pectin, starch, modified starch, cellulose, modified cellulose, carrageenan, gum Arabic, acacia gum, xanthan gum, locust bean gum and guar gum are preferred.

25

The coating may also contain one or more conventional coating materials, preferably materials which are soluble or dispersible in water. Conventional coating materials are described e.g. in WO 89/08694, WO 89/08695, EP 270608 B1 and/or WO 00/01793. Other examples of conventional coating materials may be found in US 4,106,991, EP 170360, EP 304332, EP 304331, EP 458849, EP 458845, WO 97/39116, WO 92/12645A, WO 89/08695, WO 89/08694, WO 87/07292, WO 91/06638, WO 92/13030, WO 93/07260, WO 93/07263, WO 96/38527, WO 96/16151, WO 97/23606, US 5,324,649, US 4,689,297, EP 206417, EP 193829, DE 4344215, DE 4322229 A, DD 263790, JP 61162185 A and/or JP 58179492.

30

The coating may comprise materials selected from binders, fibers, salts, water insoluble minerals, pigments, enzyme stabilizers, waxes or combinations thereof.

In a particular embodiment the coating may comprise:

- 5 - Enzyme stabilising agents. Enzyme stabilising or protective agents such as conventionally used in the field of granulation may be elements of the coating. Stabilising or protective agents may fall into several categories: alkaline or neutral materials, reducing agents, antioxidants and/or salts of first transition series metal ions. Each of these may be used in conjunction with other protective agents of the same or different categories. Examples of alkaline protective agents are alkali metal silicates, carbonates or bicarbonates which provide a chemical scavenging effect by actively neutralising e.g. oxidants. Examples of reducing protective agents are salts of sulfite, thiosulfite, thiosulfate or  $\text{MnSO}_4$  while examples of antioxidants are methionine, butylated hydroxytoluene (BHT) or butylated hydroxyanisol (BHA). Most preferred agents are salts of thiosulfates, e.g. sodium thiosulfate or methionine. Also enzyme stabilizers may be borates, borax, formates, di- and tricarboxylic acids and reversible enzyme inhibitors such as organic compounds with sulfhydryl groups or alkylated or arylated boric acids. Examples of boron based stabilizer may be found in WO 96/21716, whereas a preferred boron based stabilizer is 4-Formyl-Phenyl-Boronic Acid or derivatives thereof described in WO 96/41859 both disclosed incorporated herein by reference. Still other examples of useful enzyme stabilizers are gelatine, casein, Poly vinyl pyrrolidone (PVP) and powder of skimmed milk. The amounts of protective agent in the coating may be 5-40% w/w of the coating, preferably 5-30%, e.g. 10-20%.

- Solubilising agents. The solubility of the coating is especially critical in cases where the coated particle is a component of detergent formulation. As is known by the person skilled in the art, many agents, through a variety of methods, serve to increase the solubility of formulations, and typical agents known to the art can be found in national *Pharmacopeia*'s. Thus, the core particle may optionally comprise any agent that serves to enhance the solubility of the coated particle.
- 10 - Inorganics, such as water soluble and/or insoluble inorganic salts such as finely ground alkali sulphate, alkali carbonate and/or alkali chloride, clays such as kaolin (e.g. Speswhite™, English China Clay), bentonites, talcs, zeolites, calcium carbonate, and/or silicates.
- 15 - Binders, e.g. binders with a high melting point or indeterminately high melting points and of a non-waxy nature, e.g. polyvinyl pyrrolidone, dextrans, polyvinylalcohol, cellulose derivatives, for example hydroxypropyl cellulose, methyl cellulose or CMC. A suitable binder is a carbohydrate binder such as Glucidex 21D™ available from Roquette Freres, France.
- Fibre materials such as pure or impure cellulose in fibrous form. This can be sawdust, pure fibrous cellulose, cotton, or other forms of pure or impure fibrous cellulose. Also, 25 filter aids based on fibrous cellulose can be used. Several brands of cellulose in fibrous form are on the market, e.g. CEPO™ and ARBOCELL™. Pertinent examples of fibrous cellulose filter aids are is Arbocel BFC200™ and Arbocel BC200™. Also synthetic fibres may be used as described in EP 304331 B1 and typical fibres may be made of polyethylene, 30 polypropylene, polyester, especially nylon, polyvinyl-formate, poly(meth)acrylic compounds.

- Cross-linking agents such as enzyme-compatible surfactants, e.g. ethoxylated alcohols, especially ones with 10 to 80 ethoxy groups. These may both be found in the coating and in the core particle.
- 5 - Suspension agents, mediators (for boosting bleach action upon dissolution of the particle in e.g. a washing application) and/or solvents may be incorporated in the coating.
- Viscosity regulating agents. Viscosity regulating agents  
10 may be present in the coating.

The coating should encapsulate the core particle by forming a continuous homogenous layer.

The coating may perform any of a number of functions in the  
15 particle or granule, depending on the intended use. Thus, for example, a coating may achieve one or more of the following effects:

- (i) further reduction of the dust-formation tendency of a core particle;
- 20 (ii) further protection of active(s) in the core particle against oxidation by bleaching substances/systems (e.g. perborates, percarbonates, organic peracids and the like);
- (iii) dissolution at a desired rate upon introduction of the particle into a liquid medium (such as an aqueous medium);
- 25 (iv) provide a better physical strength of the particle of the invention.

The coating may further comprise one or more of the following: Chlorine scavengers, plasticizers, pigments, lubricants (such as surfactants or antistatic agents) and  
30 fragrances.

Plasticizers useful in coating layers in the context of the present invention include, for example: polyols such as sugars, sugar alcohols, or polyethylene glycols (PEGs) having a

molecular weight less than 1000; urea, phthalate esters such as dibutyl or dimethyl phthalate; and water.

Suitable pigments include, but are not limited to, finely divided whiteners, such as titanium dioxide or kaolin, coloured  
5 pigments, water soluble colorants, as well as combinations of one or more pigments and water soluble colorants.

As used in the present context, the term "lubricant" refers to any agent which reduces surface friction, lubricates the surface of the granule, decreases tendency to build-up of static  
10 electricity, and/or reduces friability of the granules. Lubricants can also play a related role in improving the coating process, by reducing the tackiness of binders in the coating. Thus, lubricants can serve as anti-agglomeration agents and wetting agents.

15 Examples of suitable lubricants are lower polyethylene glycols (PEGs) and ethoxylated fatty alcohols.

In a preferred embodiment of the invention the core particle of the invention is coated with a protective coating having a high constant humidity such as described in the Danish  
20 patent application WO 00/01793 pages 5-9 and given examples, which is hereby incorporated by reference.

In a further preferred embodiment the coating is substantially free of active. The term "substantially enzyme free" as used herein about a coating means that there less than  
25 5 mg of active per gram coating.

In a still further preferred embodiment the coating is thicker than known coatings in that  $D_T/D_C$  is at least 1.1, preferably at least 1.5, more preferably at least 2, more preferably at least 2.5, more preferably at least 3, most  
30 preferably at least 4.  $D_T/D_C$  is however preferably below about 100, preferably below about 50, more preferably below 25, and most preferably below 10. A most preferred range for  $D_G/D_C$  is about 4 to about 6.

The preferred thickness of the coating is at least 25  $\mu\text{m}$ . A more preferred thickness is at least 50  $\mu\text{m}$  such as at least 75  $\mu\text{m}$ , at least 100  $\mu\text{m}$ , least 150  $\mu\text{m}$ , least 200  $\mu\text{m}$ , least 250  $\mu\text{m}$  or most preferably at least 300  $\mu\text{m}$ .

5 The coating, in certain embodiments, can comprise several coating layers, each with a special function.

In a preferred embodiment the coating has an outer layer of a liquid lubricant. The purpose of the lubricant is to grease the granule so as to increase flow ability of the granule and to  
10 further inhibit dust formation when individual granules collide during handling. The lubricant is preferably a mineral oil or a nonionic surfactant, and more preferably the lubricant is not miscible with the other shell materials.

The coating material including the gas phase component  
15 should have a true density below 0.8  $\text{g/cm}^3$ , preferably below 0.6  $\text{g/cm}^3$ , more preferably below 0.4  $\text{g/cm}^3$  and most preferably below 0.2  $\text{g/cm}^3$ ,

#### **The core particle**

The core particle contains the active. Besides of the  
20 active the core particle may be constructed in any way or of any material which provides the desired functional properties of the core material, e.g. the core may consist of materials which allows readily release of the active (s) upon introduction to an aqueous medium. In one preferred embodiment the core particle  
25 is constructed of a particulate carrier (I) with the active absorbed and/or an enzyme containing layer (II) applied on the carrier surface, optionally comprising a protecting reducing agent. There may even be additional coating within the core material providing desired functional properties of the core  
30 material. Another preferred core particle is the so called T-granulate wherein active and granulation material is mixed to form granules incorporating the enzyme distributed throughout the core such as described in US 4,106,991 e.g. Example 1. Any



conventional methods and non-active materials may be used to prepare the core particle. Examples of known conventional cores particles and materials is, *inter alia*, described in, US 4,106,991 (in particular), EP 170360, EP 304332, EP 304331, EP 5 458849, EP 458845, WO 97/39116, WO 92/12645, WO 89/08695, WO 89/08694, WO 87/07292, WO 91/06638, WO 92/13030, WO 93/07260, WO 93/07263, WO 96/38527, WO 96/16151, WO 97/23606, US 5,324,649, US 4,689,297, EP 206417, EP 193829, DE 4344215, DE 4322229 A, DD 263790, JP 61162185 A, JP 58179492.

10 As a particularly preferred embodiment of the invention the core particle may be prepared by applying a layer of active onto a "placebo" carrier (active-free carrier) according to the methodology described in US 4,106,991. Optionally additional active may be absorbed into the surface of the carrier.

15 In a particular embodiment of the invention the core particle may also comprise a protective agent as described for the coating, *vide supra*, preferably mixed with the active in suitable amounts such as 0.1- 1% w/w of the coated particle, preferably 0.1-0.5% w/w, e.g. 0.33% w/w.

20 I a preferred embodiment the core particle comprises an active dispersed in a visco-elastic liquid matrix having a  $\eta'$  and a  $\eta''$  both between  $10^6$  to  $10^{14}$  measured in a cone-and-plate rheometer at 25°C and a sinusoidal frequencies  $\omega$  of 1 Hz.

25

The dry visco-elastic liquid making up the matrix wherein the active and optionally other useful components may in principle be any material or mixtures of materials which meets the requirements for viscosity and elasticity set for the visco-  
30 elastic core particles.

Preferred materials are dry organic visco-elastic materials such as dry liquid materials within organic polymers and/or monomers. Materials such as carbohydrate polymers (e.g. pectins), proteins (e.g.) gelatin, sugars, glucose sirups,

modified vegetable oils or mixtures thereof can be brought or formulated into a dry liquid state having visco-elastic properties as required in the present invention.

Preferably a majority of the components constituting the  
5 visco-elastic liquid matrix are water soluble.

In a preferred embodiment the visco-elastic liquid matrix has a  $\eta'$  and a  $\eta''$  between  $10^8$  to  $10^{12}$ , most preferably between  $10^9$  to  $10^{11}$  Pa when measured in a cone-and-plate rheometer at 25°C and a sinusoidal frequencies  $\omega$  of 1 Hz.

10 In this context the term "liquid" as used in the context of this invention is to be understood as a property of a material. A liquid material is defined as a material for which a certain amount of stress, i.e. force pr. unit area is obtained, when subjecting a deformation force, i.e. a strain, is applied to the  
15 liquid material **as long as deformation occurs**. As soon as deformation stops the stress level decreases immediately to the steady state level, which always will be exactly zero. Liquids incapable of sustaining a permanent stress in the liquid.

The term "visco-elastic liquid" as used in the context of  
20 this invention is to be understood as a property of the liquid. ---  
--- A liquid (fluid) is visco-plastic when the time span for the stress in the material to reach exactly zero after a deformation is sufficiently large. Visco-elastic liquids may be described using a simple model containing two parameters  $\eta'(\omega)$  and  
25  $\eta''(\omega)$ , which may easily be measured in a cone-and-plate rheometer (eg. Bohlin Rheometer) for different sinusoidal frequencies  $\omega$ .  $\eta'(\omega)$  may be interpreted as the elasticity of the used visco-elastic fluids and  $\eta''(\omega)$  as the viscosity. The tangent of ratio  $\delta = \eta'(\omega)/\eta''(\omega)$ , measured in degrees is  
30 called the loss tangent =  $\tan \delta$ . This definition is acknowledged in the art e.g. in Bird R.B., Armstrong R.C., Hassager O. "Dynamics of polymeric liquids", Volume 1: Fluid mechanics, John Wiley and Sons, Chapter 6, especially example 6.1.2.1 pp 281,

1977. Visco-elastic liquids of the invention has a  $\eta'$  and a  $\eta''$  between  $10^6$  to  $10^{14}$  when measured in a cone-and-plate rheometer (eg. Bohlin Rheometer) using a sinusoidal frequencies  $\omega$  of 1 Hz at 25°.

5 The term "dry" as used in the context of the present invention is to be understood as materials containing no or at least very little water. A dry solid composition or a solid may contain some water, e.g. as bound in solid crystals or it may contain water absorbed from a humid environment. The amount of water in  
10 a dry solid composition will therefore depend on the solids in the composition, the hygroscopicity of the solids and the humidity of the surrounding environment. Likewise, a dry liquid is a liquid containing no or at least very little water. Accordingly, materials which contains below 10% w/w water,  
15 preferably below 5% w/w water, more preferably below 3% w/w, more preferably below 1% w/w or no detectable amount of water are all considered dry materials.

The core particle should preferably be less than 700  $\mu\text{m}$  or 600  $\mu\text{m}$ , preferably between 50 and 500  $\mu\text{m}$ , such as between 100  
20 and 400  $\mu\text{m}$ , most preferably between 200 and 300  $\mu\text{m}$ .

In general the core particles have a preferred true density below 3  $\text{g}/\text{cm}^3$ , preferably below 2  $\text{g}/\text{cm}^3$ , more preferably below 1.5  $\text{g}/\text{cm}^3$ .

## 25 Actives

The active of the invention may any active component or mixture of active components which benefits from being separated from the liquid phase of a liquid composition. The term "active" is meant to encompass all components which upon release from the  
30 visco-elastic liquid matrix upon applying the particle of the invention in a process serves a purpose of improving the process. Suitable actives are those which are either subjects of deactivation and/or causing deactivation to other components in

the compositions of the invention. As said the active is preferably present dispersed as discrete solid particles in the visco-elastic liquid matrix.

The active may be inorganic of nature such as bleach  
5 components as mentioned *infra* or organic. Preferred actives are active biological materials which are usually very sensitive to the surrounding environment and benefits from being embedded in a visco-elastic liquid matrix, such as materials obtainable from microorganisms. Most preferred actives are peptides or  
10 polypeptides such as enzymes.

The enzyme in the context of the present invention may be any enzyme or combination of different enzymes. Accordingly, when reference is made to "an enzyme" this will in general be understood to include combinations of one or more enzymes.

15 It is to be understood that enzyme variants (produced, for example, by recombinant techniques) are included within the meaning of the term "enzyme". Examples of such enzyme variants are disclosed, e.g., in EP 251,446 (Genencor), WO 91/00345 (Novo Nordisk), EP 525,610 (Solvay) and WO 94/02618 (Gist-Brocades  
20 NV).

The enzyme classification employed in the present specification with claims is in accordance with *Recommendations (1992) of the Nomenclature Committee of the International Union of Biochemistry and Molecular Biology*, Academic Press, Inc.,  
25 1992.

Accordingly the types of enzymes which may appropriately be incorporated in granules of the invention include oxidoreductases (EC 1.-.-.-), transferases (EC 2.-.-.-),  
hydrolases (EC 3.-.-.-), lyases (EC 4.-.-.-), isomerases (EC 5.-  
30 .-.-) and ligases (EC 6.-.-.-).

Preferred oxidoreductases in the context of the invention are peroxidases (EC 1.11.1), laccases (EC 1.10.3.2) and glucose oxidases (EC 1.1.3.4)]. An Example of a commercially available oxidoreductase (EC 1.-.-.-) is Gluzyme™ (enzyme available from

Novo Nordisk A/S). Further oxidoreductases are available from other suppliers. Preferred transferases are transferases in any of the following sub-classes:

- 5 a) Transferases transferring one-carbon groups (EC 2.1);
- b) transferases transferring aldehyde or ketone residues (EC 2.2); acyltransferases (EC 2.3);
- c) glycosyltransferases (EC 2.4);
- d) transferases transferring alkyl or aryl groups, other than
- 10 methyl groups (EC 2.5); and
- e) transferases transferring nitrogenous groups (EC 2.6).

A most preferred type of transferase in the context of the invention is a transglutaminase (protein-glutamine  $\gamma$ -glutamyltransferase; EC 2.3.2.13).

- 15 Further examples of suitable transglutaminases are described in WO 96/06931 (Novo Nordisk A/S).

Preferred hydrolases in the context of the invention are: Carboxylic ester hydrolases (EC 3.1.1.-) such as lipases (EC 3.1.1.3); phytases (EC 3.1.3.-), e.g. 3-phytases (EC 3.1.3.8)

20 and 6-phytases (EC 3.1.3.26); glycosidases (EC 3.2, which fall within a group denoted herein as "carbohydrases"), such as  $\alpha$ -amylases (EC 3.2.1.1); peptidases (EC 3.4, also known as proteases); and other carbonyl hydrolases].

In the present context, the term "carbohydrase" is used to

25 denote not only enzymes capable of breaking down carbohydrate chains (e.g. starches or cellulose) of especially five- and six-membered ring structures (i.e. glycosidases, EC 3.2), but also enzymes capable of isomerizing carbohydrates, e.g. six-membered ring structures such as D-glucose to five-membered ring

30 structures such as D-fructose.

Carbohydrases of relevance include the following (EC numbers in parentheses):

$\alpha$ -amylases (EC 3.2.1.1),  $\beta$ -amylases (EC 3.2.1.2), glucan 1,4- $\alpha$ -glucosidases (EC 3.2.1.3), endo-1,4-beta-glucanase (cellulases,

EC 3.2.1.4), endo-1,3(4)- $\beta$ -glucanases (EC 3.2.1.6), endo-1,4- $\beta$ -xylanases (EC 3.2.1.8), dextranases (EC 3.2.1.11), chitinases (EC 3.2.1.14), polygalacturonases (EC 3.2.1.15), lysozymes (EC 3.2.1.17),  $\beta$ -glucosidases (EC 3.2.1.21),  $\alpha$ -galactosidases (EC 3.2.1.22),  $\beta$ -galactosidases (EC 3.2.1.23), amylo-1,6-glucosidases (EC 3.2.1.33), xylan 1,4- $\beta$ -xylosidases (EC 3.2.1.37), glucan endo-1,3- $\beta$ -D-glucosidases (EC 3.2.1.39),  $\alpha$ -dextrin endo-1,6- $\alpha$ -glucosidases (EC 3.2.1.41), sucrose  $\alpha$ -glucosidases (EC 3.2.1.48), glucan endo-1,3- $\alpha$ -glucosidases (EC 3.2.1.59), glucan 1,4- $\beta$ -glucosidases (EC 3.2.1.74), glucan endo-1,6- $\beta$ -glucosidases (EC 3.2.1.75), arabinan endo-1,5- $\alpha$ -L-arabinosidases (EC 3.2.1.99), lactases (EC 3.2.1.108), chitosanases (EC 3.2.1.132) and xylose isomerases (EC 5.3.1.5).

Examples of commercially available proteases (peptidases) include Kannase™, Everlase™, Esperase™, Alcalase™, Neutrase™, Durazym™, Savinase™, Pyrase™, Pancreatic Trypsin NOVO (PTN), Bio-Feed™ Pro and Clear-Lens™ Pro (all available from Novo Nordisk A/S, Bagsvaerd, Denmark).

Other commercially available proteases include Maxatase™, Maxacal™, Maxapem™, Opticlean™ and Purafect™ (available from Genencor International Inc. or Gist-Brocades).

Examples of commercially available lipases include Lipoprime™ Lipolase™, Lipolase™ Ultra, Lipozyme™, Palatase™, Novozym™ 435 and Lecitase™ (all available from Novo Nordisk A/S).

Other commercially available lipases include Lumafast™ (*Pseudomonas mendocina* lipase from Genencor International Inc.); Lipomax™ (*Ps. pseudoalcaligenes* lipase from Gist-Brocades/Genencor Int. Inc.; and *Bacillus* sp. lipase from Solvay enzymes. Further lipases are available from other suppliers.

Examples of commercially available carbohydrases include Alpha-Gal™, Bio-Feed™ Alpha, Bio-Feed™ Beta, Bio-Feed™ Plus,

Bio-Feed™ Plus, Novozyme™ 188, Celluclast™, Cellusoft™, Ceremyl™, Citrozym™, Denimax™, Dezyme™, Dextrozyme™, Finizym™, Fungamyl™, Gamanase™, Glucanex™, Lactozym™, Maltogenase™, Pentopan™, Pectinex™, Promozyme™, Pulpzyme™, 5 Novamyl™, Termamyl™, AMG™ (Amyloglucosidase Novo), Maltogenase™, Sweetzyme™ and Aquazym™ (all available from Novo Nordisk A/S). Further carbohydrases are available from other suppliers.

The amount of active, such as enzyme (calculated as pure 10 enzyme protein) in a core particle of the invention will typically be in the range of from about 20% to 65% by weight of the core particle, preferably no less than 25%, such as no less than 30%, 35%, 40%, 45%, 50%, 55%, 60% or 65% by weight .

#### **Method of preparing coated particles**

##### 15 Preparing core particles

Methods for preparing core particles are disclosed in the above mentioned references. Methods for preparing the dry visco-elastic liquid core particles should always contain the step of dispersing the active and optionally other materials, preferably 20 in a dry solid particulate form, in a dry visco-elastic liquid matrix to a preferably homogenous dispersion. This step could suitably be conducted at elevated temperatures to gain a lower viscosity than that of the finished particles. However, due care should be taken in not damaging the active by the heat. In this 25 context an important feature is that the visco-elastic liquid matrix is dry, because the absence of water may make it possible to apply higher temperatures than if water was present without significantly damaging the active.

After preparing this mixture a number of different step can 30 be applied to prepare liquid particles from the dispersion.

In one embodiment the dispersion is cooled to below the glass transition temperature of the visco-elastic liquid matrix

and the dispersion is crushed and/or grinded to obtain small particles of the frozen liquid.

In another embodiment the dispersion is processed while the visco-elastic liquid matrix is in its liquid state by rolling the dispersion into a thin sheet of visco-elastic liquid material and cutting out pieces. This method resembles the way Italian pasta is made by preparing sheets of material and slicing pieces of a desired shape from the sheet.

In a third embodiment the dispersion is extruded through a small hole and cut into small pieces

The particles obtained in the above mentioned is preferably rounded to achieve spherical or near spherical shape of the particles. This can be done conventionally, such as in a Marumarizer. It is however preferred to use liquid nature of the particles to shape to particles. By fluidising the particles in a fluid bed dryer and subjecting the particles to heat the surface tension of the visco-elastic liquid matrix will cause the particles to adapt a spherical shape. This procedure also facilitates any subsequent coating step because application of the coating may be conducted after rounding of the particles, but while the particles are still fluidised. In a fluid bed coating process the fluidised core particles are sprayed with a solution containing the coating material(s), and the coating is deposited on the surface of the core particles by evaporating the solution solvent.

#### Applying the Coating

Applying a coating comprising a gas phase component to a core particle may be obtained in several ways. One preferred way is to mix a gas into a coating material, e.g. by intensive mixing or by bubbling the gas through the liquid coating material, at a temperature in which the coating material is in a liquid form and then spraying the liquid coating material onto the core particles under cooling conditions so that the coating material



deposits and solidifies on the core particles thereby entrapping the gas component in the coating. This method also applies when incorporating light spheres into the coating material.

Another preferred way to provide a coating comprising a gas  
5 phase component is to prepare a coating material comprising a gas generating component, applying this coating to the core particle and treating the coated particle so as to generate a gas from the gas generating component. In a preferred embodiment the treatment is physical such as heating. In a further  
10 preferred embodiment a relatively volatile component, such as lower alkyls e.g. isopentane is mixed into the coating and by subjecting the coated particle to heat, e.g. such as in a pasteurisation process heating only the coating, the volatile component will boil and create gas filled bubbles in the  
15 coating, which will be fixed or entrapped when the coating solidifies at ambient temperature. This type of coating may also be made with carbohydrate polymers, by boiling the water contained in the polymer (pop corn popping reaction).

Another type of treatment is chemical treatment. In a  
20 preferred embodiment a compound which undergoes a chemical reaction by the treatment releasing a gas is mixed into the coating and by subjecting the coated particle to the chemical treatment the gas is formed. In a specific embodiment a preferred compound is a bicarbonate salt which is mixed into the  
25 coating material, the coating is applied to the core particle and the coated particles is treated by an acid whereby carbon dioxide gas is formed, entrapped in the solid coating.

The coating may be applied to the core particles using any conventional coating method such as in a mixer or in a fluid  
30 bed.

**Compositions comprising the coated particle and their application**

The invention also relates to compositions comprising the coated particles of the invention. The composition may be any  
5 composition, but preferred compositions are those intended for such in the food, baking and/or detergent industry. Accordingly the composition may be a food, bakers flour, dough or detergent composition or an additive to be incorporated in such compositions. Also the invention encompasses the use of the  
10 composition, e.g. for improving foodstuffs such as bread or for cleaning an object such as a cellulose containing fabric.

**Baking**

In a special embodiment of the invention we have found that our development of coated particles comprising an active is useful  
15 in baking industry.

Within the flour mill and the baking industry the use of actives, such as enzymes, is well established. Accordingly the invention provides baking compositions comprising the coated particles of the invention, in particular dough improver  
20 compositions or flour compositions comprising the dough improver.

When using enzymes in the baking industry certain enzyme activities are preferred. Flour has varying content of amylases leading to differences in the baking quality. Addition of  
25 amylases can be necessary in order to standardize the flour. Amylases and pentosanases generally provide sugar for the yeast fermentation, improve the bread volume, retard retrogradation, and decrease the staling rate and stickiness that results from pentosan gums. Examples of carbohydrases is given below.

30 Certain maltogenic amylases can be used for prolonging the shelf life of bread for two or more days without causing gumminess in the product. Selectively modifies the gelatinized starch by cleaving from the non-reducing end of the starch

molecules, low molecular weight sugars and dextrans. The starch is modified in such a way that retrogradation is less likely to occur. The produced low-molecular-weight sugars improve the baked goods water retention capacity without creating the intermediate-length dextrans that result in gumminess in the finished product. The enzyme is inactivated during bread baking, so it can be considered a processing aid which does not have to be declared on the label.

The bread volume can be improved by fungal  $\alpha$ -amylases which further provide good and uniform structure of the bread crumb.

Said  $\alpha$ -amylases are endoenzymes that produce maltose, dextrans and glucose. Cereal and some bacterial  $\alpha$ -amylases are inactivated at temperatures above the gelatinization temperature of starch, therefore when added to a wheat dough it results in a low bread volume and a sticky bread interior. Fungamyl has the advantage of being thermolabile and is inactivated just below the gelatinization temperature.

Enzyme preparations containing a number of pentosanase and hemi-cellulase activities can improve the handling and stability of the dough, and improves the freshness, the crumb structure and the volume of the bread.

By hydrolysing the pentosans fraction in flour, it will lose a great deal of its water-binding capacity, and the water will then be available for starch and gluten. The gluten becomes more pliable and extensible, and the starch gelatinize more easily. Pentosanases can be used in combination with or as an alternative to emulsifiers.

### Detergents

The coated particles of the invention may also preferably be added to and thus become a component of a detergent composition.

The detergent composition of the invention may for example be formulated as a hand or machine laundry detergent composition

including a laundry additive composition suitable for pre-treatment of stained fabrics and a rinse added fabric softener composition, or be formulated as a detergent composition for use in general household hard surface cleaning operations, or be  
5 formulated for hand or machine dishwashing operations.

In a specific aspect, the invention provides a detergent additive comprising the coated particles of the invention. The detergent additive as well as the detergent composition may comprise one or more other enzymes such as a protease, a lipase,  
10 a cutinase, an amylase, a carbohydrase, a cellulase, a pectinase, a mannanase, an arabinase, a galactanase, a xylanase, an oxidase, e.g., a laccase, and/or a peroxidase.

In general the properties of the chosen enzyme(s) should be compatible with the selected detergent, (i.e. pH-optimum,  
15 compatibility with other enzymatic and non-enzymatic ingredients, etc.), and the enzyme(s) should be present in effective amounts.

Proteases: Suitable proteases include those of animal, vegetable or microbial origin. Microbial origin is preferred. Chemically modified or protein engineered mutants are included. The  
20 protease may be a serine protease or a metallo protease, preferably an alkaline microbial protease or a trypsin-like protease. Examples of alkaline proteases are subtilisins, especially those derived from *Bacillus*, e.g., subtilisin Novo, subtilisin Carlsberg, subtilisin 309, subtilisin 147 and  
25 subtilisin 168 (described in WO 89/06279). Examples of trypsin-like proteases are trypsin (e.g. of porcine or bovine origin) and the *Fusarium* protease described in WO 89/06270 and WO 94/25583.

Examples of useful proteases are the variants described in  
30 WO 92/19729, WO 98/20115, WO 98/20116, and WO 98/34946, especially the variants with substitutions in one or more of the following positions: 27, 36, 57, 76, 87, 97, 101, 104, 120, 123, 167, 170, 194, 206, 218, 222, 224, 235 and 274.

Preferred commercially available protease enzymes include Alcalase™, Savinase™, Primase™, Duralase™, Esperase™, and Kannase™ (Novo Nordisk A/S), Maxatase™, Maxacal™, Maxapem™, Properase™, Purafect™, Purafect Oxp™, FN2™, and FN3™  
5 (Genencor International Inc.).

Lipases: Suitable lipases include those of bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Examples of useful lipases include lipases from Humicola (synonym Thermomyces), e.g. from *H. lanuginosa* (T.  
10 *lanuginosus*) as described in EP 258 068 and EP 305 216 or from *H. insolens* as described in WO 96/13580, a *Pseudomonas* lipase, e.g. from *P. alcaligenes* or *P. pseudoalcaligenes* (EP 218 272), *P. cepacia* (EP 331 376), *P. stutzeri* (GB 1,372,034), *P. fluorescens*, *Pseudomonas* sp. strain SD 705 (WO 95/06720 and WO  
15 96/27002), *P. wisconsinensis* (WO 96/12012), a *Bacillus* lipase, e.g. from *B. subtilis* (Dartois et al. (1993), Biochemica et Biophysica Acta, 1131, 253-360), *B. stearothermophilus* (JP 64/744992) or *B. pumilus* (WO 91/16422).

Other examples are lipase variants such as those described  
20 in WO 92/05249, WO 94/01541, EP 407 225, EP 260 105, WO 95/35381, WO 96/00292, WO 95/30744, WO 94/25578, WO 95/14783, WO 95/22615, WO 97/04079 and WO 97/07202.

Preferred commercially available lipase enzymes include Lipolase™ and Lipolase Ultra™ (Novo Nordisk A/S).

25 Amylases: Suitable amylases ( $\alpha$  and/or  $\beta$ ) include those of bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Amylases include, for example,  $\alpha$ -amylases obtained from *Bacillus*, e.g. a special strain of *B. licheniformis*, described in more detail in GB 1,296,839.

30 Examples of useful amylases are the variants described in WO 94/02597, WO 94/18314, WO 96/23873, and WO 97/43424, especially the variants with substitutions in one or more of the following positions: 15, 23, 105, 106, 124, 128, 133, 154, 156,

181, 188, 190, 197, 202, 208, 209, 243, 264, 304, 305, 391, 408, and 444.

Commercially available amylases are Duramyl™, Termamyl™, Fungamyl™ and BAN™ (Novo Nordisk A/S), Rapidase™ and Purastar™  
5 (from Genencor International Inc.).

Cellulases: Suitable cellulases include those of bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Suitable cellulases include cellulases from the genera *Bacillus*, *Pseudomonas*, *Humicola*, *Fusarium*, *Thielavia*,  
10 *Acremonium*, e.g. the fungal cellulases produced from *Humicola insolens*, *Myceliophthora thermophila* and *Fusarium oxysporum* disclosed in US 4,435,307, US 5,648,263, US 5,691,178, US 5,776,757 and WO 89/09259.

Especially suitable cellulases are the alkaline or neutral  
15 cellulases having colour care benefits. Examples of such cellulases are cellulases described in EP 0 495 257, EP 0 531 372, WO 96/11262, WO 96/29397, WO 98/08940. Other examples are cellulase variants such as those described in WO 94/07998, EP 0 531 315, US 5,457,046, US 5,686,593, US 5,763,254, WO 95/24471, WO  
20 98/12307 and PCT/DK98/00299.

Commercially available cellulases include Celluzyme™, and Carezyme™ (Novo Nordisk A/S), Clazinase™, and Puradax HA™ (Genencor International Inc.), and KAC-500(B)™ (Kao Corporation).

25 Peroxidases/Oxidases: Suitable peroxidases/oxidases include those of plant, bacterial or fungal origin. Chemically modified or protein engineered mutants are included. Examples of useful peroxidases include peroxidases from *Coprinus*, e.g. from *C. cinereus*, and variants thereof as those described in WO  
30 93/24618, WO 95/10602, and WO 98/15257.

Commercially available peroxidases include Guardzyme™ (Novo Nordisk A/S).

The detergent enzyme(s) may be included in a detergent composition by adding separate additives containing one or more enzymes, or by adding a combined additive comprising all of these enzymes. A detergent additive of the invention, i.e. a separate  
5 additive or a combined additive, may be formulated so as to contain one or more of the particles of the invention comprising different enzymes.

The detergent composition of the invention may be in any convenient dry form, e.g., a bar, a tablet, a powder, a granule,  
10 a paste or a liquid. A liquid detergent is preferably a dry (non-aqueous) liquid detergent.

The detergent composition comprises one or more surfactants, which may be non-ionic including semi-polar and/or anionic and/or cationic and/or zwitterionic. The surfactants are  
15 typically present at a level of from 0.1% to 60% by weight.

When included therein the detergent will usually contain from about 1% to about 40% of an anionic surfactant such as linear alkylbenzenesulfonate, alpha-olefinsulfonate, alkyl sulfate (fatty alcohol sulfate), alcohol ethoxysulfate,  
20 secondary alkanesulfonate, alpha-sulfo fatty acid methyl ester, alkyl- or alkenylsuccinic acid or soap.

When included therein the detergent will usually contain from about 0.2% to about 40% of a non-ionic surfactant such as alcohol ethoxylate, nonylphenol ethoxylate, alkylpolyglycoside,  
25 alkyldimethylamineoxide, ethoxylated fatty acid monoethanolamide, fatty acid monoethanolamide, polyhydroxy alkyl fatty acid amide, or N-acyl N-alkyl derivatives of glucosamine ("glucamides").

The detergent may contain 0-65 % of a detergent builder or complexing agent such as zeolite, diphosphate, triphosphate,  
30 phosphonate, carbonate, citrate, nitrilotriacetic acid, ethylenediaminetetraacetic acid, diethylenetriaminepentaacetic acid, alkyl- or alkenylsuccinic acid, soluble silicates or layered silicates (e.g. SKS-6 from Hoechst).

The detergent may comprise one or more polymers. Examples are carboxymethylcellulose, poly(vinylpyrrolidone), poly(ethylene glycol), poly(vinyl alcohol), poly(vinylpyridine-N-oxide), poly(vinylimidazole), polycarboxylates such as 5 polyacrylates, maleic/acrylic acid copolymers and lauryl methacrylate/acrylic acid copolymers.

The detergent may contain a bleaching system which may comprise a  $H_2O_2$  source such as perborate or percarbonate which may be combined with a peracid-forming bleach activator such as 10 tetraacetylenediamine or nonanoyloxybenzenesulfonate. Alternatively, the bleaching system may comprise peroxyacids of e.g. the amide, imide, or sulfone type.

The enzyme(s) of the detergent composition of the invention may be stabilized using conventional stabilizing agents, 15 e.g., a polyol such as propylene glycol or glycerol, a sugar or sugar alcohol, lactic acid, boric acid, or a boric acid derivative, e.g., an aromatic borate ester, or a phenyl boronic acid derivative such as 4-formylphenyl boronic acid, and the composition may be formulated as described in e.g. WO 92/19709 and 20 WO 92/19708.

The detergent may also contain other conventional detergent ingredients such as e.g. fabric conditioners including clays, foam boosters, suds suppressors, anti-corrosion agents, soil-suspending agents, anti-soil redeposition agents, dyes, 25 bactericides, optical brighteners, hydrotropes, tarnish inhibitors, or perfumes.

It is at present contemplated that in the detergent compositions any enzyme, may be added in an amount corresponding to 0.01-100 mg of enzyme protein per litre of wash liquor, 30 preferably 0.05-5 mg of enzyme protein per litre of wash liquor, in particular 0.1-1 mg of enzyme protein per litre of wash liquor.



The enzyme of the invention may additionally be incorporated in the detergent formulations disclosed in WO 97/07202 which is hereby incorporated as reference.

PATENT CLAIMS

1. A coated particle comprising a coating and a core particle comprising an active, wherein the coating comprises a gas phase component.

5

2. The particle of claim 1, wherein the gas phase component constitutes at least 20 % by volume of the coating material.

3. The particle of claim 2, wherein the coating material  
10 including the gas phase component has a true density below 0.8 g/cm<sup>3</sup>.

4. The particle of any preceding claim, wherein the gas phase component is confined within a light sphere.

15

5. The particle of any preceding claim, wherein the coating further comprises materials selected from waxes, polypeptides, and carbohydrate polymers.

20 6. The particle of claim 5, wherein the wax is a poly ethylene glycol.

7. The particle of claim 5, wherein the polypeptide is selected from gelatine, collagen, casein, chitosan, poly aspartic acid  
25 and poly glutamic acid.

8. The particle of claim 5, wherein the carbohydrate polymer is selected from pectin, starch, modified starch, cellulose, modified cellulose, carrageenan, gum Arabic, acacia gum, xanthan  
30 gum, locust bean gum and guar gum.

9. The particle of any claim preceding claims, wherein the gas phase component is selected from atmospheric air, carbon dioxide, nitrogen, noble gases.

5 10. A method for preparing the coated particle of claims 1-9 comprising a step selected from

(a) providing a coating material comprising a gas phase component and applying the gas containing coating material to a core particle or

10 (b) providing a coating material comprising a gas generating component, applying the coating material to a core particle and treating the coated particles so as to generate a gas from the gas generating component.

15 11. The method of claim 10, comprising step (b).

12. The method of claims 11, wherein the gas generating component is a volatile component and the treatment is heating.

20 13. The method of claim 11, wherein the gas generating component is bicarbonate and the treatment is an acid treatment.

14. A composition comprising the particles of claims 1-9.

25 15. The composition of claim 14, characterised by being a detergent composition further comprising a surfactant.

16. The composition of claim 14, characterised by being a dough composition further comprising a flour.

30

17. Use of the composition of claim 15, for cleaning an object.

18. Use of the composition of claim 16 for improving a dough.

ABSTRACT

Title: Particles containing a elastic coating

The present invention relates to coating materials for coating  
5 particles containing an active. Said coating materials are  
elastic and light and serve both the purpose of regulating  
density of the particles a protects the particle from damage by  
being able of absorbing impacts in the particle. The invention  
also relates to processes for the manufacture of such coated  
10 particles and to the use of such coated particles in a number of  
applications.